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DEVELOPMENT OF BATTERY SEPARATOR MATERIAL PROCESS

by

L. M. Adams
W. W. Harlowe, Jr.

QUARTERLY REPORT

SwRI Project No. 01-2015
Contract No. 951718

Prepared for

Jet Propulsion Laboratory
California Institute of Technology
4800 Oak Grove Drive
Pasadena, California 91103

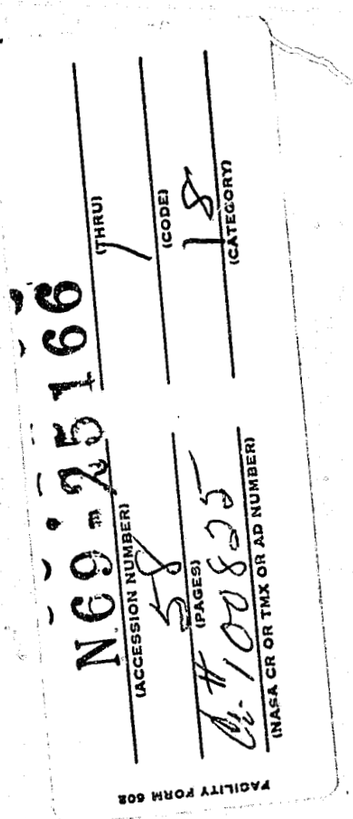
Attn: H. E. Patterson
Senior Contract Negotiator
Mail Station 190-212

April 10, 1969

Approved:

John T. Goodwin

John T. Goodwin, Director
Department of Chemistry and
Chemical Engineering



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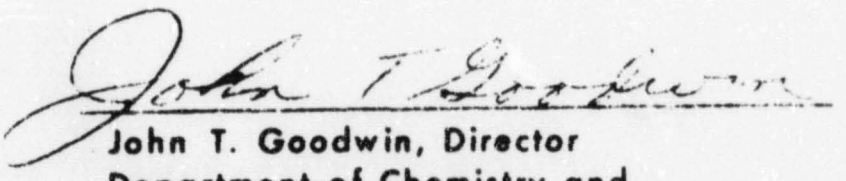

John T. Goodwin, Director
Department of Chemistry and
Chemical Engineering

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I. SUMMARY

The polyethylene film supplied by the Jet Propulsion Laboratory from material on hand has proven to be inferior to film obtained from another source. As a consequence, the Jet Propulsion Laboratory is now supplying Dow 400 polyethylene film which contains 1000 ppm of calcium carbonate as the only additive.

The new polyethylene film grafts more readily than the previously used polyethylene film and permits the use of grafting solutions containing less acrylic acid. The lower acrylic acid concentration reduces the exotherm encountered during grafting and simplifies scale-up of the grafting procedure. Battery separator material prepared from the new polyethylene film has excellent clarity and has a uniformly low electrical resistance.

Of the interlayer materials evaluated, cotton cheesecloth is the best from a standpoint of ease of handling, improved grafting of the polyethylene film, and cost.

The addition of certain acetylenic compounds minimizes homopolymer precipitation from the grafting solution. Calcium naphthenate has no effect on homopolymer precipitation, but it does improve the uniformity of the grafted film.

Polyethylene film can be readily grafted with aqueous acrylic acid and methacrylic acid solutions if a gelation inhibitor is added to

the solutions. Potassium ferricyanide and potassium ferrocyanide prevent gelation and can be readily washed from the grafted films. Work to date with aqueous acrylic acid solutions has resulted in over-grafting of the film. The electrical resistance of the grafted film is uniform and very low.

Polyethylene film has been cografted with acrylic acid and sodium vinyl sulfonate to produce a product which is uniform and of very low electrical resistance.

II. INTRODUCTION

Work has continued on the preparation of an improved sterilizable battery separator material for silver oxide-zinc batteries. To date, the only properties which have been determined on these materials are resistance to sterilization, dimensional changes, tensile strength, elongation, and electrical resistance. Battery separator materials of uniformly low electrical resistance have been prepared. The other physical properties have exceeded specifications outlined by the Jet Propulsion Laboratory.

Ion migration studies are to be determined for promising battery separator materials.

This report covers work performed during the period from January 8, 1969 through March 31, 1969.

III. EXPERIMENTAL

A. Polyethylene Film Studies

During the optimization of the acrylic acid grafting procedure, polyethylene film (1 mil) supplied by the Jet Propulsion Laboratory from stock on hand was utilized. The results obtained varied throughout the 10,000-foot rolls and between the 10,000-foot rolls. Because of this, films from several different polyethylene resins were evaluated. The films were prepared by the Freeport Laboratories of the Dow Chemical Company and were free of additives.

Results obtained on the acrylic acid grafting of some of these films in 30-foot rolls are tabulated in Tables 1-4. For comparative purposes, typical results obtained with polyethylene originally furnished by the Jet Propulsion Laboratory are presented in Table 5. The electrical resistance of the grafted Dow polyethylene resins is much more uniform than that obtained with the Jet Propulsion Laboratory supplied film.

Previous work has shown that lowering the acrylic acid concentration in the grafting solution resulted in very nonuniform grafting with the Jet Propulsion Laboratory film. Lowering the concentration of acrylic acid in the grafting solution to as low as 15 weight percent with most of the Dow polyethylene films resulted in satisfactory grafting. Dow 710M film grafted satisfactorily at 20 weight percent acrylic acid

(Table 6) and at 15 weight percent acrylic acid (Table 7). With 15 weight percent acrylic acid in the grafting solution, the Dow 110E was borderline. The other Dow films which were discussed in previous reports behaved as did the Dow 710M film.

A sample of 2-mil Dow 400 polyethylene film from a commercial run was obtained from the Dow's Findlay, Ohio plant. This material contained 1000 ppm of calcium carbonate which appeared to have no effect on the grafting procedure. The electrical resistance (Table 10) throughout four 30-foot rolls grafted in separate containers under identical ambient conditions was very uniform and ranged from 10 to 17 milliohm-inch^2 with an average value of 13 milliohm-inch^2.

Crosslinking of the above grafted film with divinylbenzene increased the resistance slightly and produced a battery separator material of excellent strength and uniformity. The data obtained before and after sterilization are presented in Table 11.

Because of the excellent results obtained with the 2-mil Dow 400 film and the fact that JPL's supply of polyethylene film had been depleted, it was recommended that future work be conducted with 1-mil Dow 400 film. JPL obtained a quantity of this film from Dow's Fresno, California plant and has supplied Southwest Research Institute with this film for future work. The results of a typical grafting run with this new material are presented in Table 12.

B. Study of Interlayer Material

In preparing rolls of polyethylene film for grafting, the film is backed with paper toweling and is rolled onto 1/4-inch aluminum pipe. The interlayer which is formed by the backing material is necessary to keep the polyethylene film separated and permit the grafting solution to reach all of the surface of the polyethylene film. Changes in the source of paper toweling sometimes caused a change in the results obtained in grafting of the polyethylene film.

A study made with various interlayer materials indicates that cloth is superior to paper toweling. Two of the best materials evaluated from a standpoint of effect on grafting and convenience in use are cotton cheesecloth and coarse nylon mesh. These interlayer materials permit the polyethylene film to be exposed to a greater volume of grafting solution and remove less of the acrylic acid through grafting of the interlayer material than does paper toweling. It was possible to obtain more uniform grafting and lower electrical resistances using these two interlayer materials with the original JPL polyethylene film (Tables 13 and 14).

When using JPL polyethylene film with a cheesecloth interlayer, it is possible to lower the acrylic acid content of the grafting solution to as low as 15 weight percent and still obtain uniformly grafted film as indicated by the electrical resistance (Table 15). At 10 weight percent acrylic acid, nonuniform grafting occurs.

With the new 1-mil Dow 400 polyethylene film, satisfactory grafting is obtained at an acrylic acid concentration as low as 10 weight percent in the grafting solution. The results obtained in a series of runs varying the acrylic acid concentration from 25 to 10 weight percent are presented in Tables 16 through 18.

C. Scale-Up Studies

Previous studies on scale-up of the grafting procedure have been conducted with both 600 and 100-foot rolls of polyethylene film. All of the previous work was with the original JPL polyethylene film, paper toweling interlayer, and an acrylic acid concentration of 25 weight percent. Excessive exotherm was frequently encountered, and the high temperatures experienced made it difficult to unroll the grafted film. When the grafted film was recovered, it frequently was opaque. It is believed that the opaqueness was caused by crosslinked acrylic acid homopolymer which could not be washed from the grafted film.

The reactor is a cylindrical chamber fabricated from aluminum sheet. The inside diameter is 12-1/2 inches, and the height is 30 inches. The wall thickness of the cylindrical portion is 1/8 inch. The reactor can be used without or with a cooling coil in the center of the reactor.

The use of a cooling coil in the reactor lowered the maximum temperature obtained in the free grafting solution, but it did now lower the temperature within the rolls of polyethylene film sufficiently to form

a product which could be easily recovered. Data from a run using the cooling coil with a grafting solution containing 25 weight percent acrylic acid are presented in Table 19. The electrical resistance is uniformly low, but the film is somewhat opaque.

As mentioned previously, the substitution of cheesecloth for paper toweling as the interlayer permits lowering of the acrylic acid concentration in the grafting solution. At this lower concentration, the maximum temperature within the rolls is less than 100°F when using a cooling coil in the reactor and is usually between 140° and 150°F without the cooling coil. The grafted film is easily recovered and has excellent clarity. The lower temperature (less than 100°F) results in nonuniform grafting of the JPL film (Table 20). The higher temperature (Table 21) produces somewhat more uniform film but not as uniform as that obtained with Dow 560E polyethylene film (Table 22).

The electrical resistance of grafted Dow 400 (1 mil) polyethylene film was uniformly low when prepared at either temperature (Tables 23 and 24).

The film washing machine which was constructed was utilized to wash one of the rolls of film from Sample No. 338. The effect of leaving the film in the washing machine for prolonged periods of time was found to be slight (Table 25). The electrical resistance of the machine washed film was somewhat higher than the film processed in the laboratory.

The cheesecloth used in the aforementioned work was purchased from a local chemical supply house and was of very open weave. A roll of a much closer weave cheesecloth (Chicopee No. 44) which was purchased from The Apparel Mart, Dallas, Texas, was found to be much easier to handle and was excellent as an interlayer material. The electrical resistance of the grafted film was uniformly low (Table 26).

D. Other Unsaturated Acids for Grafting

Several different types of polyethylene film were grafted with methacrylic acid (Tables 27-29). The effect of methacrylic acid concentration in the grafting solution is illustrated by the data in Table 29. In all cases, grafted film with low electrical resistance was obtained.

E. Additives for Grafting Solution

A number of additives have been evaluated previously for the prevention of homopolymer precipitation from the grafting solution. The most effective was iron naphthenate, as it completely prevented homopolymer precipitation. Cobalt and nickel naphthenates were effective in reducing the amount of homopolymer precipitated. As there is some question as to how completely the metal salts can be removed from the grafted film and their presence is detrimental to battery performance, other materials were evaluated for the prevention of homopolymer precipitation.

The addition of acetylenic compounds to grafting solutions which do not contain chain terminator (carbon tetrachloride) decreases the

the amount of homopolymer precipitated. The outer three feet of the film showed intermittent grafting, and the remainder of the roll exhibited uniformly low electrical resistance (Tables 30 and 31). When carbon tetrachloride is present, the usual amount of homopolymer precipitates.

The presence of calcium naphthenate in the grafting solution does not affect homopolymer precipitation, but the uniformity of the electrical resistance of the grafted JPL film was improved considerably (Table 32).

F. Aqueous Grafting Systems

Polyethylene film is readily radiation grafted in aqueous acrylic acid solutions to yield a material of very low electrical resistance, but the homopolymer forms a tenacious gel which makes it extremely difficult to recover the grafted film. It has been reported previously that the addition of ferrous sulfate to the aqueous acrylic acid solution prevents gelation, and the grafted film can be recovered readily. It has been found that potassium ferricyanide and potassium ferrocyanide will also prevent gelation of the aqueous grafting solution. These materials can be washed completely from the grafted film, and there is no danger of iron contamination of the product. The grafted films have uniform and very low electrical resistance (Tables 33-35). These samples were overgrafted as they tore into pieces during the hot water rinse. Lower concentrations of acrylic acid in the grafting solution should prevent this.

An advantage of aqueous grafting solutions is the elimination of the toxicity and fire hazard encountered with the benzene grafting solutions.

Attempts to graft polyethylene film with sodium vinyl sulfonate have been unssuccessful. However, polyethylene film has been successfully grafted with mixtures of sodium vinyl sulfonate and acrylic acid. The electrical resistance of the product is uniformly low (Tables 36 and 37). Infrared spectra of the grafted films indicate the presence of sulfonic acid groups.

Aqueous methacrylic acid grafting solutions behave similarly to aqueous acrylic acid solutions (Tables 38 and 39).

G. Ion Migration Studies

Equipment is being assembled for the determination of migration of silver, zincate, and hydroxyl ions through battery separator materials.

IV. CONCLUSIONS

Dow 400 polyethylene film is a good base stock for preparing sterilizable battery separator material with excellent uniformity as measured by the electrical resistance.

Cheesecloth and nylon mesh are superior to paper toweling as the interlayer material. Cheesecloth has been chosen because of its cost advantage.

Aqueous grafting solutions appear promising if overgrafting can be prevented.

Battery separator material containing both carboxyl and sulfonic acid groups can be prepared by cografting polyethylene film with acrylic acid and sodium vinyl sulfonate.

V. FUTURE WORK

Scale-up studies of the grafting procedure will be continued, and preparation of the remainder of the 10,000-foot order of battery separator material will be started.

Grafting of polyethylene film with methacrylic acid, acrylic acid, and sulfonic acid containing monomers will be continued.

Ion migration studies will be started.

TABLE 1. SAMPLE NO. 233
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	89°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5(1)	7, 4
15(1)	5, 5
16(2)	3, 2
28(2)	3, 1

Exotherm Data

Time to exotherm, hr:	2.0
Time to maximum exotherm; hr:	3.5
Maximum Temperature, °F:	118

Note

Prepared from Dow 710M (1 mil) polyethylene film with St. Regis paper interlayer.

(1) Neutralized and rinsed at 80°C
(2) Neutralized and rinsed at 97°C.

TABLE 2. SAMPLE NOS. 257-259
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	77°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2		
	257	258	259
5	14, 16	22, 22	14, 13
15	16, 18	19, 17	15, 16
25	16, 18	16, 16	15, 16

Exotherm Data

Time to exotherm, hr:	2.5	2.5	2.5
Time to maximum exotherm, hr:	3.5	40	4.5
Maximum temperature, °F:	100	121	106

Note

Prepared from Dow 110E (1.5 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 80°C.

TABLE 3. SAMPLE NOS. 289-290
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	289	290
5	11, 12	9, 10
15	9, 8	7, 6
25	8, 8	7, 7

Exotherm Data

Time to exotherm, hr:	2.5	2.0
Time to maximum exotherm, hr:	3.5	3.5
Maximum temperature, °F:	95	102

Note

Prepared from Dow 510M (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 97°C.

TABLE 4. SAMPLE NOS. 293-294
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	293	294
5	10, 10	9, 10
15	8, 8	8, 8
25	8, 8	9, 8

Exotherm Data

Time to exotherm, hr:	2.0	2.5
Time to maximum exotherm, hr:	3.5	4.0
Maximum temperature, °F:	77	91

Note

Prepared from Dow 560E (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 97°C.

TABLE 5. SAMPLE NO. 237
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	89°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	7, 7
15	60, 48
25	12, 36

Exotherm Data

Time to exotherm, hr:	1.0
Time to maximum exotherm, hr:	5.5
Maximum temperature, °F:	118

Note

Prepared from JPL (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 97°C.

TABLE 6. SAMPLE NO. 235
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
20 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
76 wt % Benzene	Total Dose:	0.815 Mrad
4 wt % Carbon tetrachloride	Temperature:	89°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	6, 4
15	6, 5
25	5, 6

Exotherm Data

Time to exotherm, hr:	3.0
Time to maximum exotherm, hr:	6.0
Maximum temperature, °F:	92

Note

Prepared from Dow 710M (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 97°C.

TABLE 7. SAMPLE NO. 236
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt % Benzene	Total Dose:	0.815 Mrad
3 wt % Carbon tetrachloride	Temperature:	89°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	8, 8
15	7, 10
20	6, 5

Exotherm Data

Time to exotherm, hr:	3.0
Time to maximum exotherm, hr:	6.0
Maximum temperature, °F:	94

Note

Prepared from Dow 710M (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 97°C.

TABLE 8. SAMPLE NOS. 260-261
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
20 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
76 wt % Benzene	Total Dose:	0.815 Mrad
4 wt % Carbon tetrachloride	Temperature:	77°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	260	261
5	25, 27	24, 20
15	18, 23	22, 22
25	36, 30	24, 27

Exotherm Data

Time to exotherm, hr:	2.5	2.5
Time to maximum exotherm, hr:	4.0	4.0
Maximum temperature, °F:	100	100

Note

Prepared from Dow 110E (1.5 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 80°C.

TABLE 9. SAMPLE NOS. 262-263
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt % Benzene	Total Dose:	0.815 Mrad
3 wt % Carbon tetrachloride	Temperature:	77°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	262	263
5	39, 41	118, 278
15	37, 45	45, 49
25	39, 35	39, 33

Exotherm Data

Time to exotherm, hr:	2.5	2.5
Time to maximum exotherm, hr:	4.0	4.0
Maximum temperature, °F:	98	97

Note

Prepared from Dow 110E (1.5 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 80°C.

TABLE 10. SAMPLE NOS. 282-285
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	80°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2			
	282	283	284	285
5	12, 14	14, 15	17, 16	15, 14
15	12, 13	10, 11	13, 13	10, 10
25	11, 12	12, 12	12, 13	11, 11

Exotherm Data

Time to exotherm, hr:	2.0	4.5	-	3.5
Time to maximum exotherm, hr:	4.0	5.0	-	5.5
Maximum temperature, °F:	100	84	-	100

Note

Prepared from Dow 400 (2 mil) polyethylene film with St. Regis paper interlayer. Film contained 1000 ppm calcium carbonate. Neutralized and rinsed at 97°C.

TABLE 11. SAMPLE NOS. 282-285
GRAFTED AND CROSSLINKED

A. Electrical Properties

Sample No.	Average Resistance, milliohm-inch^2		Standard Deviation		Standard Deviation % of Average	
	B.S. (1)	A.S. (2)	B.S. (1)	A.S. (2)	B.S. (1)	A.S. (2)
282	17	16	1.8	1.9	10.5	11.9
283	18	15	2.7	2.5	15.2	16.8
284	19	17	3.4	3.1	18.2	18.7
285	18	16	1.4	1.7	7.8	11.0

TABLE 11 (Continued)

B. Physical Properties

Sample No.	Average Thickness, mil			Average Width, inch			Average Length, inch			Tensile Strength[1] psi	Elongation[1] %
	Dry	B.S.[1]	A.S.[2]	Dry	B.S.[1]	A.S.[2]	Dry	B.S.[1]	A.S.[2]		
282	2.6	2.8	3.1	1.00	1.08 (8.0)	1.06 (6.0)	2.02	2.20 (8.9)	2.13 (5.5)	1576	>100
283	2.5	2.7	3.0	1.01	1.11 (9.9)	1.10 (8.9)	2.03	2.19 (7.9)	2.10 (3.4)	1598	>100
284	2.8	3.0	3.3	1.01	1.12 (10.9)	1.10 (8.9)	2.05	2.23 (8.8)	2.19 (6.8)	1586	>100
285	2.7	2.8	3.0	0.98	1.12 (14.3)	1.09 (11.2)	2.00	2.25 (12.5)	2.15 (7.5)	1524	>100

Note

Numbers in parentheses are percent change from dry dimensions.

(1) Before sterilization (B.S.) - wet with 40% KOH
(2) After sterilization (A.S.) - wet with 40% KOH

TABLE 12. SAMPLE NO. 328
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	77°F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	8, 10
15	8, 9
25	8, 8

Note

Prepared from Dow 400 (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 97°C.

TABLE 13. SAMPLE NOS. 275-276
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	75°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	275	276
5	8, 7	8, 7
15	8, 7	9, 9
25	7, 7	7, 10

Note

Prepared from JPL polyethylene film with nylon mesh interlayer. Neutralized and rinsed at 97°C.

TABLE 14. SAMPLE NO. 270
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	82 °F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	8, 8
15	8, 9
25	8, 9

Exotherm Data

Time to exotherm, hr:	5.0
Time to maximum exotherm, hr:	9.0
Maximum temperature, °F:	93

Note

Prepared from JPL polyethylene film with cheesecloth interlayer. Neutralized and rinsed at 97°C.

TABLE 15. SAMPLE NOS. 271-273
GRAFTED ONLY

Grafting Solution Composition

271	272	273	
20	15	10	wt % Acrylic acid
76	82	88	wt % Benzene
4	3	2	wt % Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	82°F
Atmosphere:	Nitrogen
Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2		
	271	272	273
5	9, 8	16, 15	36, 40
15	11, 9	17, 15	113, 45
25	11, 12	15, 15	136, 23

Exotherm Data

Time to exotherm, hr:	3.5	3.0	3.0
Time to maximum exotherm, hr:	8.0	9.0	15.0
Maximum temperature, °F:	100	99	88

Note

Prepared from JPL polyethylene film with cheesecloth interlayer. Neutralized and rinsed at 97°C.

TABLE 16. SAMPLE NOS. 329-330
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride	Temperature:	77°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	329	330
5	5, 6[2]	6, 8[1]
15	5, 6[2]	6, 7[1]
25	5, 6[2]	5, 7[2]
30	-	6, 7[2]

Exotherm Data

Time to exotherm, hr:	-	5.0
Time to maximum exotherm, hr:	-	13.5
Maximum temperature, °F:	-	90

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Neutralized and rinsed at 80°C
(2) Neutralized and rinsed at 97°C.

TABLE 17. SAMPLE NOS. 331-332
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
20 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
76 wt % Benzene	Total Dose:	0.815 Mrad
4 wt % Carbon tetrachloride	Temperature:	77°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	331	332
5	6, 8(2)	9, 10(1)
15	8, 8(2)	8, 8(1)
25	7, 7(2)	8, 8(2)
30	-	7, 8(2)

Exotherm Data

Time to exotherm, hr:	7.0	7.0
Time to maximum exotherm, hr:	14.0	15.0
Maximum temperature, °F:	100	99

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer

(1) Neutralized and rinsed at 80°C
(2) Neutralized and rinsed at 97°C.

TABLE 18. SAMPLE NOS. 333 AND 335
GRAFTED ONLY

Grafting Solution Composition			Experimental Conditions for Grafting	
333	335			
15 wt %	10 wt %	Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt %	88 wt %	Benzene	Total Dose:	0.815 Mrad
3 wt %	2 wt %	Carbon tetrachloride	Temperature:	77°F
			Atmosphere:	Nitrogen
			Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	333	335
5(1)	19, 17	15, 17
15(1)	18, 20	17, 22
25(2)	11, 12	14, 14
30(2)	16, 10	14, 15

Exotherm Data

Time to exotherm, hr:	8.5	-
Time to maximum exotherm, hr:	14.0	-
Maximum temperature, °F:	81	-

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Neutralized and rinsed at 80°C
(2) Neutralized and rinsed at 97°C.

TABLE 19. SAMPLE NO. 300
GRAFTED ONLY

Grafting Solution Composition

25 wt % Acrylic acid
70 wt % Benzene
5 wt % Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	64°F
Atmosphere:	Nitrogen
Roll Length:	100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
30	6
40	9
50	8
60	7
70	8
80	12
90	10
100	13
110	10

Exotherm Data

Thermotabs at 50-ft level in the roll indicated a temperature of 165°F.

Note

Neutralized and rinsed at 97°C. Prepared from JPL polyethylene film with cheesecloth interlayer. Used cooling coil in solution. Highly grafted with homopolymer impregnated in the film.

TABLE 20. SAMPLE NOS. 305-306
GRAFTED ONLY

Grafting Solution Composition

15 wt % Acrylic acid
82 wt % Benzene
3 wt % Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate: 0.012 Mrad/hr
Total Dose: 0.815 Mrad
Temperature: 64°F
Atmosphere: Nitrogen
Roll Length: 100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	305	306
5	51, 28	22, 25
15	26, 17	--
25	42, 59	29, 20
35	26, 15	--
45	29, 33	27, 50
55	16, 29	--
65	21, 17	30, 117
75	64, 59	--
85	67, 109	35, 43
95	255, 50	--
105	44, 28	39, 70
115	186, > 3000	139

Exotherm Data

Thermotabs at 50-ft level in the roll indicated a temperature less than 100°F.

Note

Neutralized and rinsed at 97°C. Prepared from JPL polyethylene film with cheesecloth interlayer. Film has excellent clarity. Cooling coil in solution.

TABLE 21. SAMPLE NO. 307
GRAFTED ONLY

Grafting Solution Composition

15 wt % Acrylic acid
82 wt % Benzene
3 wt % Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate: 0.012 Mrad/hr
Total Dose: 0.815 Mrad
Temperature: 64°F
Atmosphere: Nitrogen
Roll Length: 100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	23
	41
25	27
	27
45	16
	31
65	48
	41
75	293
	67

Exotherm Data

Thermotabs at 50-ft level in the roll indicated a temperature less than 150°F but more than 140°F.

Time to exotherm, hr:	16.0
Time to maximum exotherm, hr:	23.0
Maximum temperature, °F:	172

Note

Neutralized and rinsed at 97°C. Prepared from JPL polyethylene film with cheesecloth interlayer.

TABLE 22. SAMPLE NO. 309
GRAFTED ONLY

Grafting Solution Composition

15 wt % Acrylic acid
82 wt % Benzene
3 wt % Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	60°F
Atmosphere:	Nitrogen
Roll Length:	100 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	JPL 2	Dow 560E
1	13	8
	15	7
5	15	8
	12	12
25	20	7
	20	9
45	21	8
	18	9
65	21	10
	25	10
85	26	11
	25	12
95	29	12
	34	14

Exotherm Data

Thermotabs at 50-ft level indicated a temperature of 130°F in JPL 2, and more than 140°F but less than 150°F in the Dow 560E.

Time to exotherm, hr:	16.0
Time to maximum exotherm, hr:	23.0
Maximum temperature, °F:	168

Note

Neutralized and rinsed at 97°C. Prepared from 3 rolls of JPL No. 2 polyethylene film and 1 roll of Dow 560E (1 mil) polyethylene film; cheesecloth interlayer.

TABLE 23. SAMPLE NO. 337
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt % Benzene	Total Dose:	0.815 Mrad
3 wt % Carbon tetrachloride	Temperature:	60°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
10	12, 14
20	9, 12
30	10, 18
40	12, 11
50	12, 7
60	10, 11
70	14, 12
80	19, 19
90	12, 13
100	14, 13

Exotherm Data

Thermotabs at 50-ft level in the roll indicated a temperature less than 100°F.

Note

Neutralized and rinsed at 97°C. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer. Cooling coil in solution.

TABLE 24. SAMPLE NO. 338
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
82 wt % Benzene	Total Dose:	0.815 Mrad
3 wt % Carbon tetrachloride	Temperature:	60°F
	Atmosphere:	Nitrogen
	Roll Length:	100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
10	9, 10
20	10, 12
30	8, 8
40	10, 10
50	10, 10
60	8, 10
70	9, 9
80	11, 10
90	13, 10
100	10, 11

Exotherm Data

Thermotabs at 50-ft level in the roll indicated a temperature less than 150°F but more than 140°F.

Time to exotherm, hr:	14.0
Time to maximum exotherm, hr:	22.0
Maximum temperature, °F:	157

Note

Neutralized and rinsed at 97°C (Roll No. 2). Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

TABLE 25. SAMPLE NO. 338
GRAFTED ONLY

Grafting Solution Composition

15 wt % Acrylic acid
82 wt % Benzene
3 wt % Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	60°F
Atmosphere:	Nitrogen
Roll Length:	100 feet (4 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
10(1)	12, 11
20(1)	13, 13
30(1)	14, 12
40(2)	12, 12
50(2)	15, 15
60(2)	16, 16
65(2)	16, 16
75(3)	17, 17
89(3)	17, 17
95(3)	18, 18

Exotherm Data

Time to exotherm, hr:	14.0
Time to maximum exotherm, hr:	22.0
Maximum temperature, °F:	157

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer. Machine washed at 97°C (Roll No. 1).

(1) Left in 5% KOH solution over week end.
(2) 31 minutes in KOH solution and left in water over week end.
(3) 31 minutes in KOH solution and 31 minutes in water.

TABLE 26. SAMPLE NO. 356
GRAFTED ONLY

Grafting Solution Composition

15 wt % Acrylic acid
82 wt % Benzene
3 wt % Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate: 0.012 Mrad/hr
Total Dose: 0.815 Mrad
Temperature: 63°F
Atmosphere: Nitrogen
Roll Length: 100 feet (5 rolls in reactor)

Electrical Properties

Footage	Resistance, milliohm-inch^2
10	7, 9
20	7, 8
30	9, 9
40	8, 9
50	9, 9
60	9, 9
70	9, 9
80	7, 8
90	10, 9
100	7, 8

Exotherm Data

Time to exotherm, hr: 6.0
Time to maximum exotherm, hr: 18.0
Maximum temperature, °F: 113

Note

Prepared from Dow 400 (1 mil) polyethylene film with Chicopee No. 44 cheesecloth interlayer. Neutralized and rinsed at 97°C. Roll No. 1 processed.

TABLE 27. SAMPLE NOS. 291-292
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Methacrylic acid	Dose Rate:	0.012 Mrad/hr
75 wt % Benzene	Total Dose:	0.815 Mrad
	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	291	292
5	6	12
	8	13
15	9	10
	8	11
25	7	11
	7	11

Exotherm Data

	291	292
Time to exotherm, hr:	2.0	2.0
Time to maximum exotherm, hr:	3.5	3.5
Maximum temperature, °F:	77	77

Note

Prepared from Dow Resin 510M (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 97°C.

TABLE 28. SAMPLE NOS. 295-296
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Methacrylic acid	Dose Rate:	0.012 Mrad/hr
75 wt % Benzene	Total Dose:	0.815 Mrad
	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2	
	295	296
5	8	10
	8	8
15	12	8
	10	9
25	11	12
	10	12

Exotherm Data

Time to exotherm, hr: Time to maximum exotherm, hr: Maximum temperature, °F:	No exotherm	No exotherm

Note

Prepared from Dow Resin 560E (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 97°C.

TABLE 29. SAMPLE NOS. 339-342
GRAFTED ONLY

Grafting Solution Composition					Experimental Conditions for Grafting	
339	340	341	342			
25	20	15	10	(wt % Methacrylic acid)	Dose Rate:	0.012 Mrad/hr
75	80	15	10	(wt % Benzene)	Total Dose:	0.815 Mrad
					Temp.:	72°F
					Atmosphere:	Nitrogen
					Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2			
	339	340	341	342
5(1)	13, 14	14, 12	14, 16	19, 18
14(1)	13, 14	12, 13	24, 26	24, 23
20(2)	8, 8	10, 10	14, 13	11, 10
25(2)	8, 7	10, 10	12, 9	14, 11

Exotherm Data

Time to exotherm, hr:	--	2.0	2.0	2.0
Time to maximum exotherm, hr:	--	4.0	4.0	4.0
Maximum temperature, °F:	--	76	76	76

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Neutralized and rinsed at 80°C.
(2) Neutralized and rinsed at 97°C.

TABLE 30. SAMPLE NO. 315
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
75 wt % Benzene plus 4.0g ethynyl cyclohexanol per 2 kilo of grafting solution	Total Dose:	0.815 Mrad
	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	8, 9
15	11, 10
25	11, 13

Exotherm Data

Time to exotherm, hr:	2.5
Time to maximum exotherm, hr:	3.5
Maximum temperature, °F:	78

Note

Neutralized and rinsed at 97°C. Prepared from Dow 560E (1 mil) polyethylene film with St. Regis paper interlayer. Small amount of homopolymer precipitated. Intermittent grafting outer 3 feet.

TABLE 31. SAMPLE NO. 316
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
75 wt % Benzene plus 4.0g Surfynol 104 per 2 kilo of grafting solution	Total Dose:	0.815 Mrad
	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
Random	11, 11
	11, 13

Exotherm Data

Time to exotherm, hr:	0.5
Time to maximum exotherm, hr:	1.5
Maximum temperature, °F:	121

Note

Neutralized and rinsed at 97°C. Prepared from Dow 560E (1 mil) polyethylene film with St. Regis paper interlayer. Small amount of homopolymer precipitated. Intermittent grafting outer 3 feet.

TABLE 32. SAMPLE NO. 274
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
70 wt % Benzene	Total Dose:	0.815 Mrad
5 wt % Carbon tetrachloride plus 20g calcium naphthenate (6%) per 2 kilo of grafting solution	Temperature:	82°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	10, 8
15	8, 11
25	8, 11

Exotherm Data

Time to exotherm, hr:	1.5
Time to maximum exotherm, hr:	4.0
Maximum temperature, °F:	95

Note

Prepared from JPL polyethylene film with St. Regis paper interlayer. Homopolymer formed. Neutralized and rinsed at 97°C.

TABLE 33. SAMPLE NO. 312
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % acrylic acid	Dose Rate:	0.012 Mrad/hr
75 wt % Water plus 9.1 g $K_4Fe(CN)_6 \cdot 3H_2O$ per 2 kilo of grafting solution	Total Dose:	0.815 Mrad
	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
Random sampling	4, 3
" "	4, 5
" "	5, 5

Note

Neutralized and rinsed at 97°C. Prepared from Dow 560E (1 mil) polyethylene film with St. Regis paper interlayer. Homopolymer precipitated but solution did not gel.

TABLE 34. SAMPLE NO. 313
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
75 wt % Water plus 7.1g $K_3Fe(CN)_6$ per 2 kilo of grafting solution	Total Dose:	0.815 Mrad
	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
Random sampling	6, 5
" "	5, 5
" "	5, 6

Exotherm Data

Time to exotherm, hr:	2.5
Time to maximum exotherm, hr:	3.5
Maximum temperature, °F:	78

Note

Neutralized and rinsed at 97°C. Prepared from Dow 560E (1 mil) polyethylene film with St. Regis paper interlayer. Homopolymer precipitated but solution did not gel.

TABLE 35. SAMPLE NO. 346
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
15 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
85 wt % Water plus 4.3g $K_3Fe(CN)_6$ per 2 kilo of grafting solution	Total Dose:	0.815 Mrad
	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
Random sampling	5, 5
" "	6, 6
" "	5, 5

Exotherm Data

Time to exotherm, hr:	2.0
Time to maximum exotherm, hr:	8.0
Maximum temperature, °F:	83

Note

Neutralized and washed at 97°C. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

TABLE 36. SAMPLE NO. 297
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
4.7 wt % Sodium vinyl sulfonate	Dose Rate:	0.012 Mrad/hr
18.8 wt % Acrylic acid	Total Dose:	0.815 Mrad
76.5 wt % Water plus 5.2g $FeSO_4 \cdot 7H_2O$ per 2 kilo of grafting solution	Temperature:	75°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	5, 3
15	6, 6
25	5, 6

Exotherm Data

Time to exotherm, hr:	4.0
Time to maximum exotherm, hr:	5.5
Maximum temperature, °F:	77

Note

Prepared from Dow Resin 560E (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and rinsed at 97°C.

TABLE 37. SAMPLE NO. 310 (Duplicate of 297)
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
4.7 wt % Sodium vinyl sulfonate	Dose Rate:	0.012 Mrad/hr
18.8 wt % Acrylic acid	Total Dose:	0.815 Mrad
76.5 wt % Water plus 5.2g	Temperature:	72°F
$FeSO_4 \cdot 7H_2O$ per 2 kilo	Atmosphere:	Nitrogen
of grafting solution	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
6	5, 6
15	6, 6
25	7, 6

Exotherm Data

Time to exotherm, hr:	2.5
Time to maximum exotherm, hr:	3.5
Maximum temperature, °F:	78

Note

Neutralized and rinsed at 97°C. Prepared from Dow 560E (1 mil) polyethylene film with St. Regis paper interlayer.

TABLE 38. SAMPLE NO. 299
GRAFTED ONLY

Grafting Solution Composition	Experimental Conditions for Grafting	
25 wt % Methacrylic acid	Dose Rate:	0.012 Mrad/hr
75 wt % Water plus 5.2g $FeSO_4 \cdot 7H_2O$ per 2 kilo of grafting solution	Total Dose:	0.815 Mrad
	Temperature:	75°F
	Atmosphere:	Nitrogen
	Roll Length:	30 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
5	5, 4
15	7, 6
25	9, 9

Exotherm Data

Time to exotherm, hr:
Time to maximum exotherm, hr:
Maximum exotherm, °F:
} No exotherm

Note

Prepared from Dow Resin 560E (1 mil) polyethylene film with St. Regis paper interlayer. Neutralized and washed at 97°C.

TABLE 39. SAMPLE NO. 345
GRAFTED ONLY

Grafting Solution Compositions	Experimental Conditions for Grafting	
25 wt % Methacrylic acid	Dose Rate:	0.012 Mrad/hr
75 wt % Water plus 7.1g $K_3Fe(CN)_6$ per 2 kilo of grafting solution	Total Dose:	0.815 Mrad
	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

Electrical Properties

Footage	Resistance, milliohm-inch^2
Random sampling	5, 4

Exotherm Data

Time to exotherm, hr:	2.0
Time to maximum exotherm, hr:	8.0
Maximum temperature, °F:	94

Note

Neutralized and washed at 97°C. Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.